Fabrication and characterization of Acetone Treated Natural Fibre Reinforced Polymer Composites

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CERTIFICATE

This is to certify that the thesis entitled "Fabrication and characterization of Reinforced Polymer composite" is submitted by Nirjharini Samal in partial fulfilment for the requirements for the award of Master of Science degree in physics department at National institute of technology, Rourkela is an authentic work carried out by her under my supervision and guidance.

To the best of my knowledge, the matter embodied in the thesis has not been submitted to any other institute/university for the award of any degree.

Place: Rourkela

Date: 9.5.2012

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ABSTRACT

The Acetone treatment of coir fibre composites were prepared using treated coir fiber and epoxy resin using handmade mold. XRD patterns confirm that degree of crystallinity increase by the treatment of coir fibre with acetone.SEM image says roughness surface structure of composites. It confirms from the SEM, the adhesion is increased after treatment. FTIR study No peak shifting has been occurred. However the peak becomes narrower in case of treated fibre due to decrease of hydroxyl group. The peak intensity has been increased

LIST OF FIGURE

CHAPTER 1

INTRODUTION

1.1. OVERVIEWS ON COMPOSITE:

The composite materials are very advent gable over conventional materials because of theirdifferent character like higher specific strength, stiffness and fatigue by which structural design to be more versatile. Composite materials consist of two or more constituents but different physically separable phases[(Krobjilowski, 33(2), 2003)].But when the composites phase material hasdifferent physical propertiesthen it is recognised as being a composites material.Composites are consists a strong load carrying material(reinforcement) which is embedded in weak material (matrix).The strength and rigidity provides by reinforced material to support the structural load whereas matrixor binder provides the position andorientation of the reinforcement.The constituents of the composites show their individualphysical and chemical properties until themtogetherely produce a combination of qualities which is impossible to produced by aindividual constituent.The reinforcement materials are plateletsor particles orfibres and they are mainly added to improve mechanical properties such as strength, stiffnessand toughness of the matrix material.

1.2. Types of composites

The composite material can be groupedinto categories depending upon theon nature of the matrix [1].fabrications method is also vary according to physical and chemical properties of the matrices and reinforcing fibres.

(***a) Metal matrix composites (MMCs)***

In metal matrix composites the matrix is metal, so it is called metal matrix composites. Aluminium, Magnesium and Titanium are examples of MMCs and carbon and silicon carbide are taken as fiber.For the design propose metals are usually reinforced .so that the elastic stiffness and strength of the metals can be increased

(b) Ceramic matrix composites (CMCs)

It consists ceramic matrix so it is called as Ceramic matrix composites .Alumina, Calcium Alumina silicate are the examples of CMCs. These are reinforced by silicon carbide. The CMC havehighstrength, hardness, highservice temp limits for ceramics, chemicalinertness forceramics, and low density. The ceramic materials are resistance to high temp, so it has a tendency to become brittle and fracture. High temp tolerance of super alloys is also offered by these composites but it has not capable to give such a high density. The fabrication of composites is difficult due to brittle nature of ceramics. In the production of the CMC material the starting materials are generally in the powder form.

The ceramicsmatrices are mainly classified as: 1.Glasswhich is easy to fabricate due to low softening temp. Exampleof glass - Borosilicate and Alumina silicate,2.Conventional ceramics like siliconnitride, aluminium oxide and zirconium oxide these are fully crystalline, 3.Cement and concreted carbon components.

(c) Polymer Matrixcomposites (PMCs)

The polymer matrix composites are very common and considered as an advanced composites. The PMCS composite consists of a polymer thermoplasticor thermosetting reinforced by fibrei.e. natural carbon and boron. Thevariety of shapes and sizes can be fashioned by these materials. Great strength and stiffness along with resistance to corrosion is also provided by this material. The PMCs composites have low cost, highstrength and simple manufacturing principles.The constituents the polymer composites often show excellent specific properties due to its low density.

1.3. Naturalfibre composites

For a long time the fibre-reinforced composites have dominate in a variety of application due to of their high specific strength and modulus.But themanufacture, use and removal of traditional fibre-reinforced plastic, mainly made of glass, carbon or aramid fibre-reinforced thermoplastic and thermo set resins are damaged seriously to the environmental.So aalternative method was found by including natural fibre composites. Naturalfibres are generallyproducedfrom animal, plantand mineral sources.

Natural fibres can be classified according to the detailed classification is shown in figure1.1.

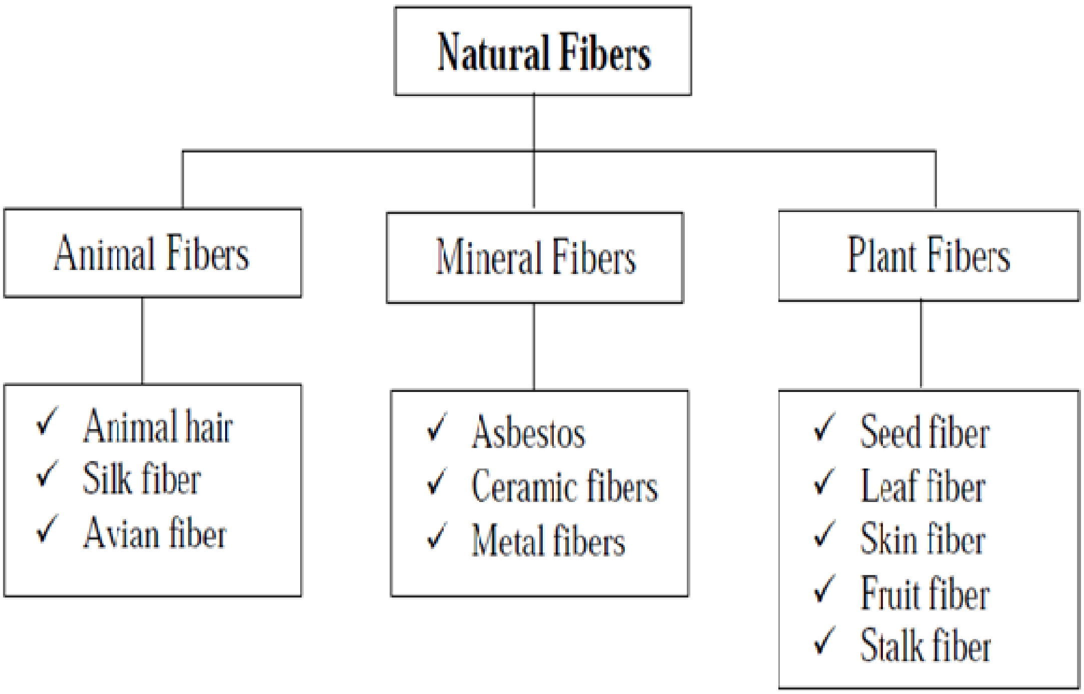


Figure 1.1 Classification of natural fibers

Animalfibre:*Animal fibre consists largelyproteins: instances are spider silk,sinew,catgut ,wool and hair such as cashmere,mohair,alpaca,angora..Animal hair taken from animals or hairy mammals.*e.g.sheep'swool, goathair, horse hair etc. And silk fibres are collected from dried saliva of bugs or insects during the preparation of cocoons. We can take example like silk from silk worms. Avianfibres are the fibres from birds, e.g. feather fiber.Animals or hairy mammals.

Mineral fibre: *Mineral fibres comprised absestors, which is only naturally occurring long fibres.*This one of the categorized of mineral fiber.other categorized are.-Variations are serpentine and amphiboles, anthophyllite.Ceramicfibres include glass fibresthat areglass ,wood and quartz.

And others are aluminiumoxide, siliconcarbides, and borocarbide.Metal fibres consist of aluminium fibres.

Plantfiber: *plantfibres are consists generally cellulose.Cotton, jute, flax, ramie, sisal and hemp are the examples of plant fibre. In the manufacture of paper and cloth cellulose fibres is required .These fibres are classified as:*

-*Seedfibre, those are collected from the seed and seed case e.g.Kapok and cotton*

- *Leaffibreisthe fibre collected from the leaves e.g.Agave and sisal.*

- *Skinfibres are collected from the skin or blast surrounding the stem of their respective plant. Banana, flax, jute, hemp and soybean are the examples of plant fibre.*

- *Fruitfibres are the fibres collected from the fruit of the plant, e.g. coconut (coir) fibre*

-*Stalk fibre are the fibres are actually the stalk of the plant.e.g straws of wheat, rice, barley.*

-*Bamboo and grass are also included in this type of fibre. wood is alsoconsidered as plant fibre.*

1.4. ADVANTAGES OF NATURAL FIBRES-:

Natural fibre has many advantages:

-It has Low specific weight that gives higher specific strength and stiffness than glass. This property is useful especially in parts designed for bending stiffness.

-Naturalfibres are renewable resource and the production required little energy.Co_2 is used while oxygen in given back to the environment.

-It can be producedwith low cost investment i.e. very used to makes the materialsas an interesting product for low wage countries.

-It has an advantage of Friendly processing, no wear of tooling, and no skin irritation.

-Thermal recycling is possible, in the other hand glass causes problems in combustion furnaces.

-Naturalfibrehas Good thermal and acoustic insulating properties.

1.5. DISADVANTAGES OF NATURAL FIBER-:

Instead of many advantages of natural fibres, there is something disadvantages, we can also found in natural fibre

-It has Lower strength properties, particularly its impact strength.

-NaturalfibreshaveVariable quality, depending on unpredictable influenced such as weather.

-It absorbsMoisture which causes swelling of fibres.

-It has lower durability, fibre treatments can improve the considerably.

-Poor fibre resistance

CHAPTER 2

LITERATURE SURVEY

The literature survey part contains some of the recent reports published on the naturalfibre based polymer composites with special emphasis on coir fibre reinforced polymer composites.

(2.1) On natural fibre

The natural fibre-reinforced composite's mechanical properties mainly dependupon manyparameters, such as fibre strength, modulus, fibre length and orientation, in addition to the fibre-matrix interfacial bond strength.

For high mechanical properties of composites a strong fibre-matrix interface is critical. For effective stress transfer from the matrix a good interfacial bond is required to the fibre by which maximum utilization of the fibre strength in the composites is achieved[2]. The Modification of the fiberby various treatmentsalso improves resistance to moisture induced degradation of the interface and the composites properties[3]. Besides this, processing techniques (conditions)have asignificant influence on the mechanical properties offibre reinforced composites[4]. The naturalfibres like flax, hemp, jute and sisal etchave very good mechanical properties and also comparable with glass fibre in specific strength and modulus[5]. On natural fibre a number of investigations have been collected.

For the evaluation of mechanical properties, the jute fabric- reinforced polyster wastested compared with wood composites. From this evalution,it was found that the jute fibre composite has better strengths than wood composites[6]. In the literature the work on banana fibre in reinforcing polymers is very rare. Corbiere-Nicollieret.al. was studied for mechanical properties of banana fibre by physically and chemically .From his study it was found that banana fibre composite has good flexural strength. Pothanet.al. is also investigated for short banana fibre; He was concentrated on the effect of fibre length and fibrecontent.The tensile and flexural properties of the green composites with different pineapplefibre content and the virgin resin were comparing by Luo and Netravali. There are also many investigation on the sisal fiber.Thisfibreis fairly coarse and inflexible. It has good strength, durability, ability to stretch, affinity for certain for dyestuffs and resistance to deterioration in seawater. Ahmed et al.investigated aresearch work on filament wound cotton fibre reinforced for reinforcing high-density polyethylene resin.The new type wood based filler was studied by Fuat et al. Came from oil palm wood flour (OPWF) composite by thermo gravimetric analysis and the outcomesare very encouraging. Influence of different surface modifications of jute on the performance of the bio composites studied Mohanty et al.

(2.2) On coir fibre reinforced composites:

Many aspects of the use fibres as reinforcement in polymer-matrix composites are described in the literature. Coir is very abundant, versatile, renewable, cheap and biodegradable lignocelluloses fibre used for making a wide variety of products [(Satyanarayana)]. Coir has been also been tested as filler or reinforced in different composite materials.Coconut coir is the most interesting products as it has the lowest thermal conductivity and bulk density .The addition of coconut coir reduced the thermal conductivity of the composite specimens and yielded a lightweight product. Development of composites materials for buildings using natural fibre as coconut coir with low thermal conductivity is an interesting alternative which would solve environment and energy concern. Coir fiber-polyster composites were tested as helmets, as roofing Coir–polyester composites with untreated and treated coir fibres, and with fibre loading of 17 wt%, were tested in tension, flexure and notched Izod impact. The results found with the untreated fibres show clear signs of the presence of a weak interface long prosefibres without a obtained. Although showing better mechanical performance, the composites with treated fibres resin adhered to the fibre- and low mechanical properties werepresent, however only moderation the values of the mechanical properties analysed. Acetylating of coir fibres increases the hydrophobic behaviour, which increases the resistance to fungi attack and also increases tensile strength of coir-polyester composites. However, thefibre loading has to be fairly high,45wt% or even higher, to attain a significant reinforcing effect. But there is no improvement in the flexural strength from these results; it is evident that the usual fibre treatment that described so far did not significantly change the mechanical performance of coir-polyester composites. Although there are several reports in the literature which discuss the mechanical behaviour of natural fibre reinforced polymer composites.The present work hasanaim todevelop a new class of natural fibre based polymer composites with Acetone treatment and to analyse their mechanical behaviour by experimental.

2.3. Objectives of the Research Work:

The objectives of my project work are

- To develop a new class of natural fiber based polymer composites to increase the potential of coir fiber.

- Development of mechanical properties and other properties of coir fiber by treatment.

CHAPTER 3

MATERIAL AND METHOD

This chapter describes the details of processing of the composites and the experimental procedures followed for their mechanical characterization. The raw materials used in this work are

1. Coconut fibre
2. Epoxy resin
3. Hardener

3.1. SPECIMEN PREPARATION

The fabrication of the various composite materials is carried out through the hand lay-up technique. Short coconut coir fibresare reinforced with epoxy LY 556 resin, chemically belonging to the ‘epoxide’ family is used as the matrix material. The common name is Bisphenol A DiglycidylEther. The low temp curing epoxy resin (Araldite LY 556) and corresponding hardener (HY951) are mixed in a ratio of 10:1 by weight as recommended. The coir fibre is collectedfrom the rural areas of Odisha.

3.2. TREATMENT OF FIBER

Fibres as received are washed with distilled water to remove the surface dirt present in the fibres and then thefibres are dried in air circulating oven at a temp of 100^0c until it gains a constant weight .Then the fibres are designed as washed fibers.For Acetone the fibres are cooked in acetone for 12h so that it attains a hohlraum character which means the substances lying in layers with free spaces in between. During this process the fibres are cooked in the solution under gradual rise and fall of the temp of the bath from 30^0 to 55.5^0c.This process of heating and cooling was done intermittently per every 2h for a period of 12h.finally, thefibre bundles are removed from the mixture at a temp of 30^0c.

3.3. FABRICATION OF COMPOSITE:

A handmade wooden mold is designed for the fabrication of the randomly oriented raw coir fibre-reinforced epoxy composite (RCFREC) and acetonefibre-reinforced epoxy composite (ACFREC). First, a releasing plastic is spread over the bottom of the wooden mold. Heavy duty silicon spray is applied to the plastic sheet for easy removal of the composite plate. The fibres are cut into 20 mm length and distributed uniformly at the bottom of the mold which is prepared before. Approximately 25 gmof the fibre is used for the fabrication of the composite. Initially, epoxy and hardener are mixed together on a weight percentage of 10:1 to form a matrix. The matrix is poured over thefibres evenly then pressed and pushed down withthe iron roller to avoid and eliminate the air bubbles. Finally, load is given to it to remove excess matrix and left for curing at room temperature for 24h.

EXPERIMENTAL TECHNIQUES

XRD: X-Ray crystallography is a method used to determining the arrangement of atoms with in a crystal in which a beam of X-rays strikes .From the angles and intensities of the diffracted beam, the 3-D picture of the density of electrons within the crystal can be studied-ray diffraction .It is a versatile, non-destructive technique that reveals details information about crystallographic structure of the material.

Principle of XRD:

Bragg's law: when a beam of parallel X-rays penetrating a stack of planes of spacing d_{hkl} (as shown in fig) at an angle of incidence θ each plane will reflects a portion of the incident beam. Then thereflected rays combine to form a diffracted beam if they differ in phase by a whole number of wavelength i.e. if the path difference AB-AD=nλ where n is an integer.

AB= d/sinθ

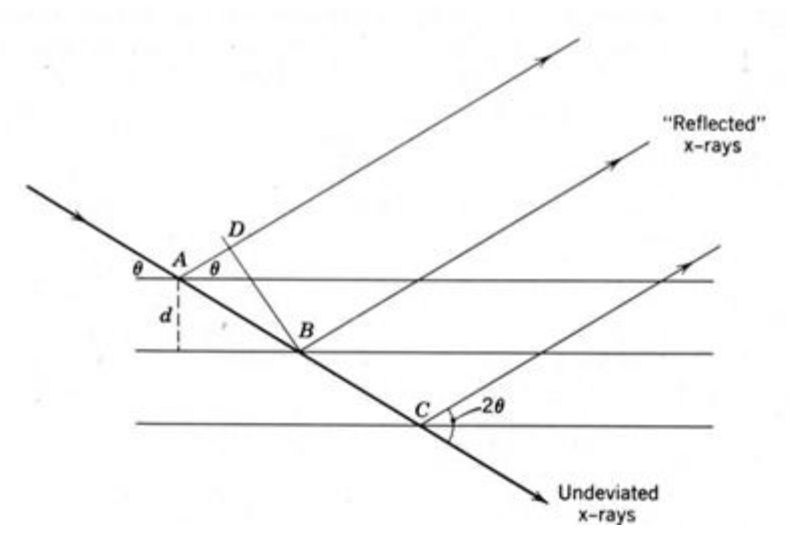
AD=AB cosθ= (d/sinθ) (cos2θ)

nλ=AB-AD=2dsinθ

2d sinθ=λ ...... (1)

Where n=1, 2, 3.....

Eqn 1 is the Bragg condition for diffraction (B.D. Cullity Elements of X-ray diffraction (1978)



- Fig2. The condition for reflection- Bragg's law

There are two geometrical factors are in the x-ray

(1) The incident beam, reflecting plane, and the diffracted beam are coplanar.

(2) The angle between the diffracted beam and the transmitted beam is 2θ. This is known as the diffraction angle.

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Fig.3.XRD machine

INSTRON:

Instron is test equipment designed to evaluate the mechanical properties of materials and components.

Hindman and George Burr in 1946, workedtogether, to determine the properties of new materials.. This led to the formation of instronEngineering Corporation.

Application:

- tensile strength testing
- compressive strength
- fatigue testing
- flexural strength testing

(^ "GEORGE BURR, 86". *Boston Globe*. 2003-11-20. Retrieved 2008-03-05)

Fig4. Instron

FTIR:

(Fourier Transform Infrared)

whenIR radiation is passed through a sample . The sample absorbed some of the infrared radiation and some of it is passed through (transmitted). This resulting spectrum represents the molecular absorption and transmissions, creating a molecular finger print of the sample.

Information provided by FTIR

- Identify unknown materials.
- The quality or consistency of a sample can determine .
- The amount of components in a mixture determine

(Introduction to Fourier Transform Infrared Spectrometry (2001 thermo NicoletCorporation alright reserved, worldwide)

SEM: (scanning electron microscope)

Using Scanning electron microscope microstructure features were studied. A scanning electron microscope images a sample by scanning it with a beam of electrons in a raster scan pattern. The sample's surface topography, composition, and electrical conductivity is also known from the SEM .

RESULT AND DISCUSSION

XRD ANALYSIS:

The XRD patterns of both Acetone and Raw coir fibres are given in figure. The full width at half maximum (FWHM) of the diffraction peaks of both raw and Acetone samples are analyzed are analyzed. The diffractograms of both Acetone and raw coir fibre display a well defined peak around $2\theta=22.45^0$ which is the characteristics of cellulose [6]. The tablebelow shows the crystalline size and degree of crystallineof Acetone and raw fibre. From the XRD pattern of Acetone and Raw fibre it can be seen that, after acetone treatment the degree of crystalline of the fibre has been increased. This change is due to the reduction of lignin content of the coir fibre and the rearrangement of cellulose chain. The changes in the crystalline peaks in the figure of Acetone sample denote the changes in the crystalline region. The increase of percent of crystalline peak area relative to the whole diffraction area suggests an improvement in the degree of crystalline of the Acetone fibre. May be due to decrease in crystal distortion and defects, the dimension and crystallites is increased [7].

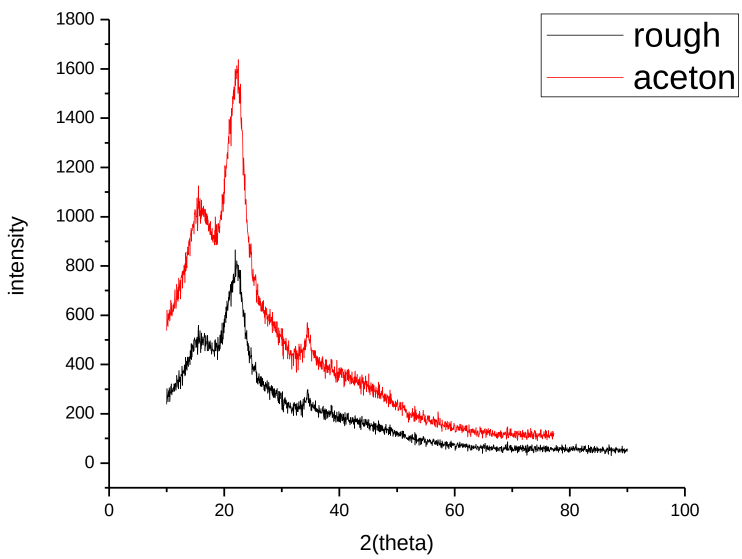


Fig-5XRD of raw and Acetone coir fibre

Table 1.The crystalline size and crystalline of raw and acetone coir fibre

Coir fibre	crystallity size	Degree of crystallinity (%)
Raw	31.64	62.5
Acetone	41.37	64.19

FLEXURAL STRENGTH:

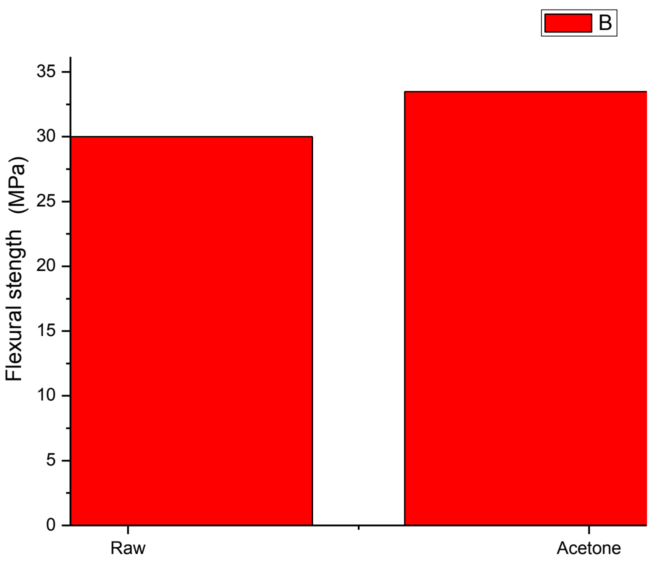
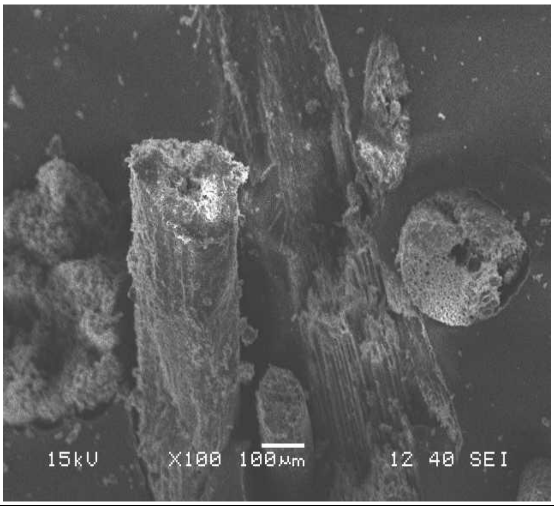


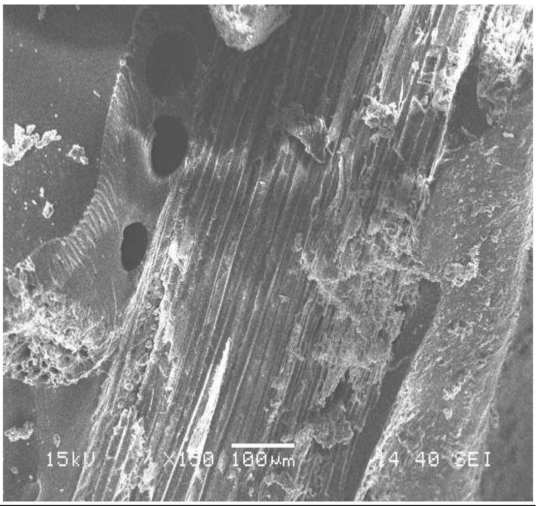
Fig-6The flexural strength of raw and Acetonecoir fibre

The figure shows the flexural strength of both raw fibre and acetone fiber.From the 3 point bend test it is found that the strength of acetone fibre is higher than raw fibre. For the fibre-reinforced composites the interfacial zone plays a leading role in transferring the load between fibre and matrix which affects the mechanical properties such as strength.[].this finding demonstrates that flexural failure depends mainly on the fibre /matrix adhesion. The increased value of flexural strength in case of acetone may be increase in effective surface area availablefor contact with the matrix [8]. The flexural strength of the coir increases after acetone treatment is due to the dissolution of hemicelluloses, development of crystalline and fibrillation thus creating superior bonding with matrix.

SEM ANALYSIS:



a



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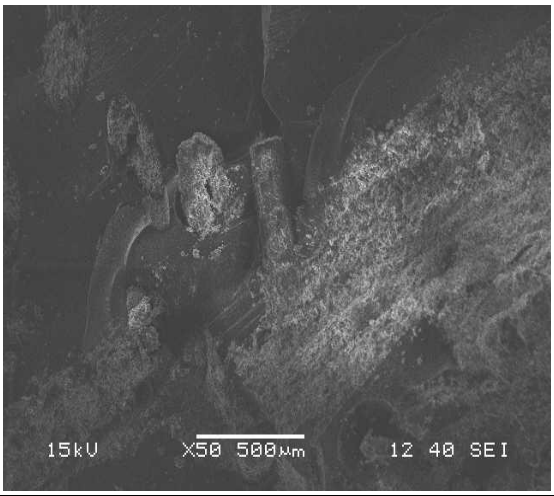

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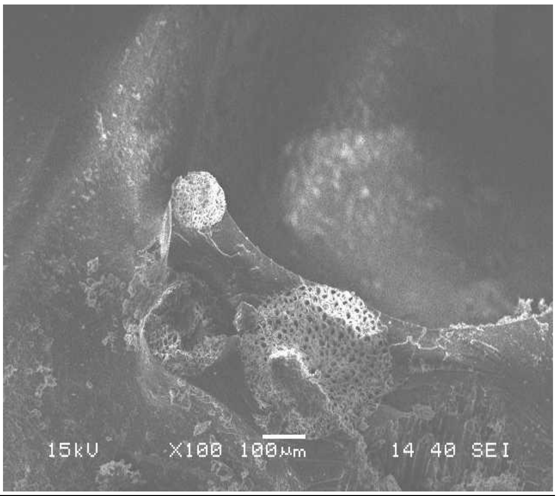

d

Fig-7 SEM of raw and Acetone fibre

The SEM images of fractured surfaces of rough (a),(c) and Acetone treated coir fibre (b),(d) are shown in the figure. It is observed that the treatment has improved the surface roughness of the fibrre as compared to the untreated fibre. From fig a it is confirmed that the adhesion between the fibre and matrix is poor, as there are gap around the fibre at the interface whereas in the treated composite the fibre matrix adhesion is shown by fibre breakage rather than fibre pullout.

FTIR ANALYSIS:

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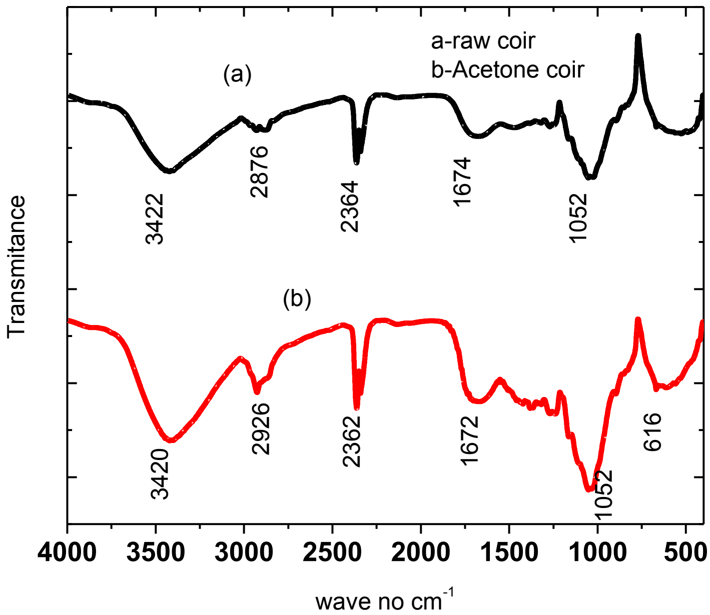

Fig-8 Ftir analysis

From the FTIR analysis the 3422 cm^{-1}peak the broad intense peak atin raw coir fiber is due to the O–H stretchingfor hydrogen-bonded hydroxyl group present in polysaccharide,however, this peak gets narrower in the case of acetone treated fiber because of the reduction of theO–H group. The weak peak which is at1674 cm-1 in raw coir fiber is due to the presence ofhemicelluloses and can be assigned to the C=O stretching.It is found that the absorbance intensity ratio of this peak decreases after acetone treatment, which suggests that acetone treatment isresponsible for the partial removal of hemicelluloses in theFiber.2876 cm^{-1}corresponds to C-H group which is increase in case of acetone treated fibre. This C-H bond is due to reinforcement of material. In acetone treated fiber the peak 1672 cm^{-1} which corresponds to water absorbed in cellulose.

Conclusion:

In this study, we synthesized the reinforced polymer and study the nature of composites by the treatment of fibre whichwas prepared with the help of coir fibreand epoxy by handmade mould. XRD patterns confirm that with the increase in the percentage of fibre in the bio composite the degree of crystalline of the composite increases. SEM micrograph shows that the treatment has improved the surface roughness of the fibrre as compared to the untreated fibre. No peak shifting has been occurred. However the peak becomes narrower in treated fibre. The peak intensity has been increased.

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